# Application of waste sorghum stem (sorghum bicolour) as a raw material for microfibre cellulose

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# Application of waste sorghum stem (sorghum bicolour) as a raw material for microfibre cellulose

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**Abstract.** Microfibre cellulose (MFC) is one of trending topics in advanced material researches. This work was aimed to study the characteristics of MFC produced from waste sorghum stem. To produce MFC, the amorphous part presence in the fibre such as hemicellulose and lignin had to be removed through chemical process. This study compared two methods for preparing the waste sorghum stem i.e. 1) Alkalization 1-Hydrolysis-Alkalization 2-Acetylation and 2) Alkalization-Bleaching-Acetylation. Thermal stability, crystallinity, morphology, and functional group analysis were the parameters measured in this research. The method that produce MFC with better quality was the first method. The MFC had a significant reduction of amorphous fraction with crystallinity index of 81.07%. From STA analysis it was found that the MFC product was able to endure the thermal treatment at 315°C without any significant weight loss.
**Keywords:** waste sorghum stem; fibre; microfibre cellulose; alkalization; bleaching

## 1. Introduction

Sorghum is mainly used as raw material for producing sugar [1]. The waste sorghum stem contains cellulose, hemicellulose, and lignin with composition around 40-44% w/w, 27-35% w/w, and 18-20% w/w respectively [2]. The natural fibre that presence in the waste sorghum stem has not been optimally utilized for producing higher value product.

Cellulose fibre receives a great deal of interest considering the ability of this material on increasing the biodegradability of a polymer. Biopolymer is the current trend especially in research regarding advanced material so that the usage of petroleum based polymer can be reduced. The cellulose fibre for biopolymer application is usually in a form of micro fibre with size <100 μm [3]. This fibre, which is known as microfibre cellulose (MFC), has the ability to reinforce the biopolymer [4]. It is crucial that the fibre must be sufficiently small in size in order to improve the mechanical strength of the biopolymer [5].

In MFC production from cellulose, the impurities such as wax and oil as well as amorphous fraction such as hemicellulose and lignin have to be removed first. There are three common methods to synthesize cellulose fibre, mechanical, chemical or enzymatic. Mechanical methods involve crushing and grinding while chemical method employs alkalization, hydrolysis, bleaching, acetylation, and another chemical treatment. Endoglucanase is commonly used in synthesizing cellulose fibre through enzymatic method [6].

Former studies synthesized cellulose from sorghum plant by combining mechanical and chemical methods [7]. This study found that alkalization with NaOH 4% will remove most of lignin thus produce 'cleaner' cellulose fibre. However the fibre size was larger than 200 μm. Another study used NaOH 20% [8]. The remaining lignin and hemicellulose was relatively very small. Bleaching treatment involve soaking the sorghum fibre from alkalization in NaClO solution 1.7% [7]. Three cycle of bleaching treatment produced cellulose fibre with better appearance and smaller size i.e. 2-4 μm.

This study used chemical method for synthesizing MFC from waste sorghum stem which consisted of several following treatments, alkalization (NaOH 5%), hydrolysis ($H_2SO_4$ 25%), bleaching (NaClO 5%) and acetylation ($CH_3OOH$ 8%). This study compared two methods for preparing the waste sorghum stem i.e. 1) Alkalization 1-Hydrolysis-Alkalization 2-Acetylation and 2) Alkalization-Bleaching-Acetylation. Thermal stability, crystallinity, morphology, and functional group analysis are the parameters measured in this research. The method that produce MFC with better quality was the combination of alkalization, bleaching and acetylation.

## *2.* Material and Methods

### *2.1. Preparation for raw material*

The waste sorghum stem was cleaned from any dirt. Then the substrate was crushed using blender and screened using 23 mesh sieve. This preparation was aimed to produce waste shorgum stem at uniform size of 710 μm.

Alkalization 1-Hydrolysis-Alkalization 2-Acetylation (Al1-Hi-Al2-As)

1. Alkalization 1

The purpose of this treatment was to remove impurities and wax from the surface of waste sorghum stem thus creating roughness in the surface. Waste sorghum stem at uniform size was soaked in NaOH 15% solution for 1 hour at 70°C. Then the substrate was washed using distilled water until it has pH=7 and dried.

2. Hydrolysis

Substrate that was produced from Alkalization 1 was soaked in $H_2SO_4$ 25% solution at room temperature for 2 hours. The purpose of this treatment to produce fibre with micro/nano size.

3. Alkalization 2

Alkalization was performed one more time by soaking the substrate in NaOH 20% solution at room temperature for 2 hours. This second alkalization was conducted to remove the remaining impurities from previous treatment.

4. Acetylation

In this treatment the fibre was soaked in acetic acid 8% at room temperature for 1 hour. The purpose of this treatment is to coat the fibre so it has hydrophobic characteristic.

Alkalization-bleaching-Acetylation (Al-B-As)

1. Alkalization

The purpose of this treatment was to remove impurities and wax from the surface of waste sorghum stem thus creating roughness in the surface. Waste sorghum stem at uniform size was soaked in NaOH 15% solution for 1 hour at 70°C. Then the substrate was washed using distilled water until it has pH=7 and dried.

2. Bleaching

This treatment had similar effect with alkalization but with stronger intensity. Substrate that was produced from previous treatment was soaked in NaOCl 5% solution at room temperature for 2 hours.

3. Acetylation

Substrate was soaked in acetic acid 8% at room temperature for 1 hour.

### *2.2 Fibre characterization*

After all fibre modification treatment has been finalized, the resulting fibre was characterized to analyze its morphology using Scanning Electron Microscope (SEM). The alteration of functional groups in the

fibre was also analyzed using Fourier Transform Infrared (FTIR). The crystallinity index of the fibre was examined using X-Ray Diffraction (XRD) while the fibre thermal stability was evaluated using Thermogravimetric Analysis (TGA).

## 3. Result and Discussion

### *3.1 Functional group analysis*

Fig. 1 presents the spectrum curve of the untreated sorghum fibre. The figure shows several peaks with wavenumber of 898 $cm^{-1}$ which indicates the chemical bond of C-O-C from β-1,4-glycosidic—the amorphous area in cellulose [9]. The peak at wavenumber of 1035 $cm^{-1}$ indicates the chemical bond of C-C and C-O from cellulose, hemicellulose and lignin in sorghum fibre. The phenolic C-O, aromatic rings, and C=C from lignin is represented by peak with wavenumber of 1244, 1514, and 1602 $cm^{-1}$ respectively. The presence of acetyl group and ester group in hemicellulose or carboxylic acid in ferulic group and p-coumeric in lignin are indicated by peak with wavenumber of 1737 $cm^{-1}$ [10]. The 2850 and 2917 $cm^{-1}$ spectrums show C-H bond while the 3338 $cm^{-1}$ is for the –OH group in cellulose, hemicellulose and lignin.

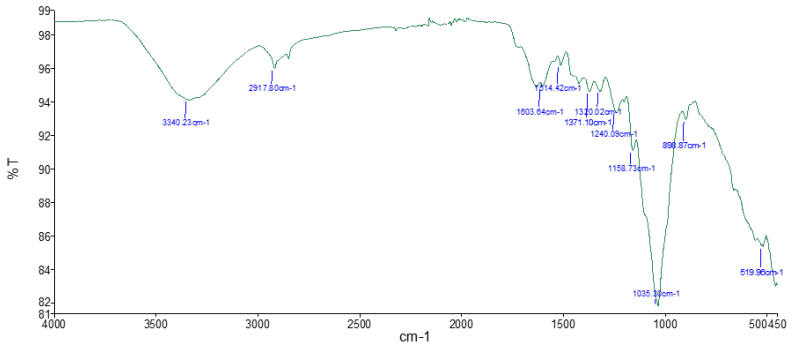


**Figure 1.** FTIR analysis results for untreated sorghum fibre.

The FTIR analysis results for Al1-Hi-Al2-As treatment and Al 15%-B-As are depicted in Fig. 2. It can be seen from the Fig. that the Al1-Hi-Al2-As treatment was better on removing hemicellulose and lignin as well as reducing the hydrophilic characteristic of the fibre. The percentage of light transmitted by functional groups that indicate hemicellulose and lignin were higher for fibre that received the Al1-Hi-Al2-As treatment, meaning that in this fibre the composition of hemicellulose and lignin were lower.

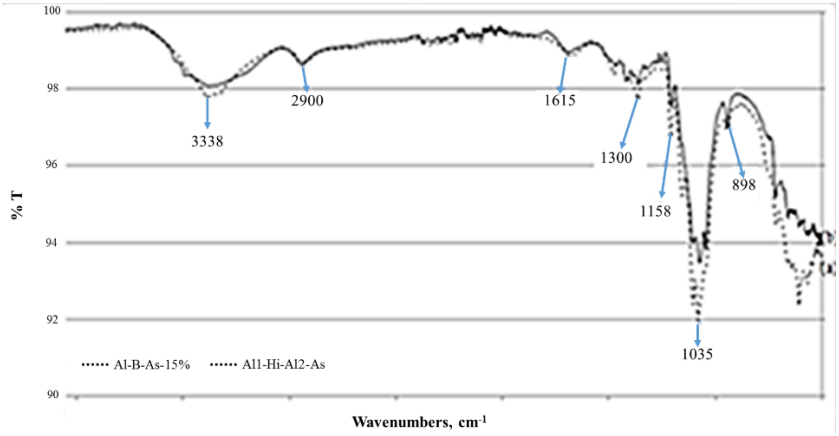


**Figure 2.** FTIR analysis results for Al1-Hi-Al2-As treatment and Al 15%-B-As.

*3.2 Fibre Morphology Analysis*

The morphology of untreated sorghum fibre is presented in Fig. 3. At magnification of 100x, Fig. 3(a) shows that fibre size is around 700 mm while in 550x of magnification we can see that the virgin sorghum fibre still has layers of lignin and hemicellulose. In addition, the cellulose fibres are also still attached to each other. Some parts of the fibre surface are peeled off which is probably lignin layers that was eroded when the fibre was crushed in blender. At larger magnification (Fig. 3(c)) we can see there are perforations in some part of the eroded fibre surface which probably the xylem area of the sorghum.

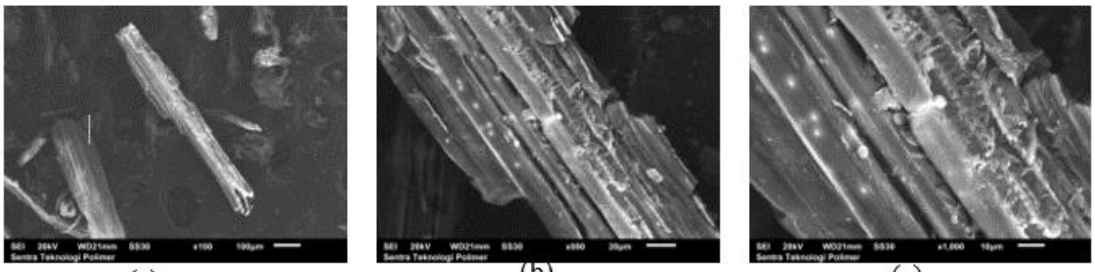


**Figure 3.** Morphology of untreated sorghum fibre with magnification of (a)100x, (b) 550x and (c) 1000x.

Fig. 4 shows the morphology of the sorghum fibre that has received Al1-Hi-Al2-As treatment. In Fig. 4(a) at 100x of magnification, it can be seen that the smallest fibre size from this treatment was 20 μm. From the Fig. 4, we can also see that most of cellulose fibre has been separated. Fig. 4(b) and (c) show that hemicellulose has been removed from the treatment. We can also observe that this treatment does not deteriorate the cellulose fibre, indicated by the absence of amorphous area on the fibre.

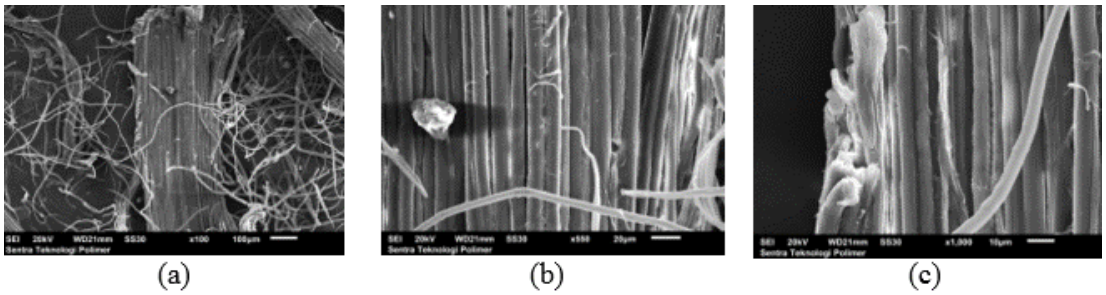


**Figure 4.** Morphology of sorghum fibre after receiving Al1-Hi-Al2-As treatment with magnification of (a) 100x, (b) 550x and (c) 1000x.

Similar results were obtained from the Al-B-As treatment. Fig. 5(a) shows that the smallest fibre size from this treatment was also 20 μm. Fig.s 5 (b) dan (c) demonstrate that most part of cellulose fibre has been detached, indicating that some of hemicellulose has been removed from the treatment.

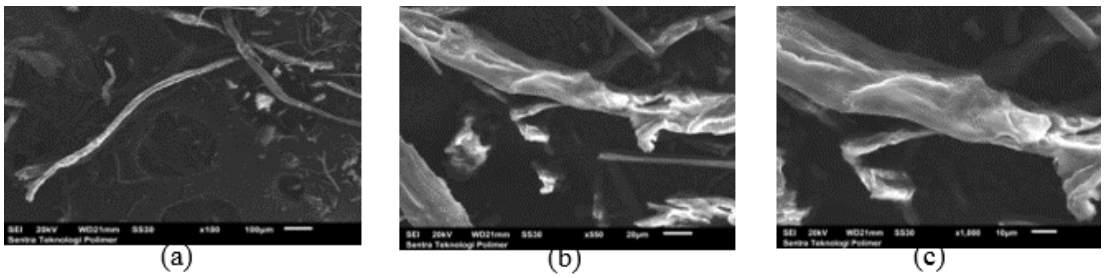


**Figure 5.** Morphology of sorghum fibre after receiving Al-B-As treatment with magnification of (a) 100x, (b) 550x and (c) 1000x.

*3.3 Thermal stability analysis*

The thermal characteristic of cellulose fibre from waste sorghum stem was studied using Thermogravimetric Analysis (TGA) at temperature range of 50-500°C. The TGA result is shown in Fig. 6. This figure shows how fibre mass change as the function of temperature. As the temperature increases from 50-300°C, the fibre mass remain constant. However when the temperature is further increased, the fibre mass is reduced significantly. The fibre from Al1-Hi-Al2-As was starting to experience weight loss at 315°C while the fibre from the other treatment was starting to degrade at lower temperature i.e. 300°C. This result shows that the Al1-Hi-Al2-As treatment produces fibre with better thermal stability compared to Al-B-As treatment.

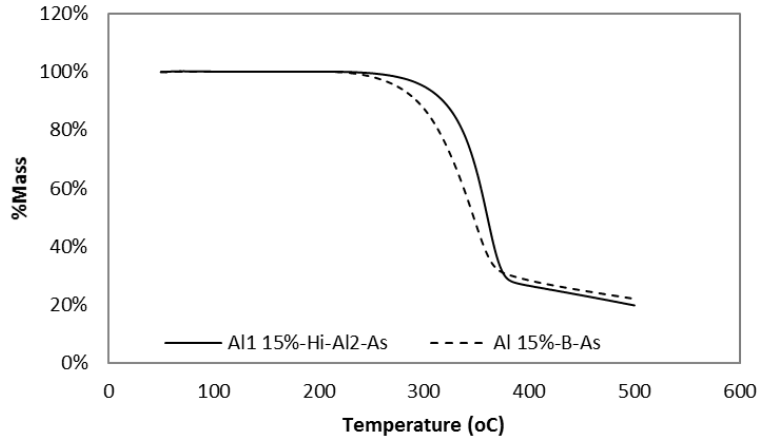


**Figure 6.** TGA result for thermal characteristic of cellulose fibre from waste sorghum stem.

*3.4 Fibre crystallinity*

The crystallinity of cellulose fibre from waste sorghum stem was measured by using XRD analysis as can be seen from Fig. 7. However XRD result only is not enough to compare the crystallinity of each sorghum fibre. Therefore the crystallinity index of each fibre should be calculated first using equation (1).

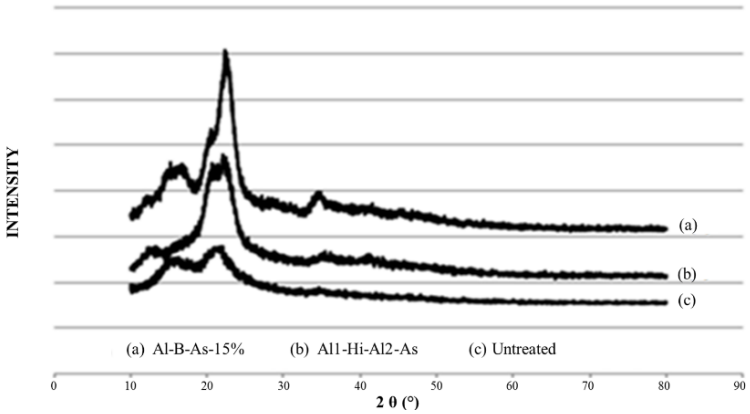


**Figure 7.** XRD analysis of fibre from waste sorghum stem; (a) and (b) with treatment, (c) untreated.

$$Crystallinity\ index = (Imax - Imin\ )/Imax \times 100\% \quad (1)$$

where I is for intensity. The calculation results for crystallinity index of each fibre are listed in table 1. The chemical treatment on sorghum fibre removes most of lignin and hemicellulose. Hence, the disappearance of amorphous fraction in the fibre exposes the semicrystralline fraction of cellulose. This phenomenon, and how different treatment produce different result can be detected by XRD analysis. Fig. 7 shows that sorghum fibre that underwent the Al-B-As treatment has the highest peak compared to the other two fibre samples. But as has been explained in previous paragraph, the height of the peak is not sufficient as an evidence to find out which sample has the highest crystallinity index.

**Table 1.** Crystallinity index of sorghum fibre.

| Treatment | Intensity | | crystallinity index (%) |
|---|---|---|---|
| | Minimum | Maximum | |
| Untreated | 148 | 256 | 42,18 |
| Al1-Hi-Al2-As | 106 | 560 | 81,07 |
| Al-B-As | 193 | 817 | 76,37 |

The low crystallinity index of sorghum fibre indicates that it has amorphous characteristic. The Al1-Hi-Al2-As treatment produced sorghum fibre with crystallinity index 81.07%. This index is higher compared to result from [8] and [11] in which the crystallinity index was 62.5% and 77% respectively.

## 4. Conclusion

Two chemical methods for synthesizing MFC from waste sorghum stem was conducted. Based on analysis results it was found that the Al1-Hi-Al2-As treatment produced MFC with higher crystallinity index compared to the Al-B-As treatment. The MFC that was produced from the former method has the smallest size of 20 μm, crystallinity index 81.07%, and able to endure thermal treatment at 315°C.

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